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Key indicators

Single-crystal X-ray study

$T = 113\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

R factor = 0.038

wR factor = 0.109

Data-to-parameter ratio = 16.8

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

3-(2-Acetoxyethyl) 5-methyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate

The title compound, $\text{C}_{20}\text{H}_{22}\text{N}_2\text{O}_8$, is an analog of nefidipine. The crystal packing is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

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Comment

4-Aryl-1,4-dihydropyridine-3,5-dicarboxylic diesters of the nefidipine type have become almost indispensable for the treatment of cardiovascular diseases since they first appeared on the market in 1975 (Yiu & Knaus, 1999; Goldmann & Stoltefuss, 1991). The title compound, (I), is a nefidipine analog.

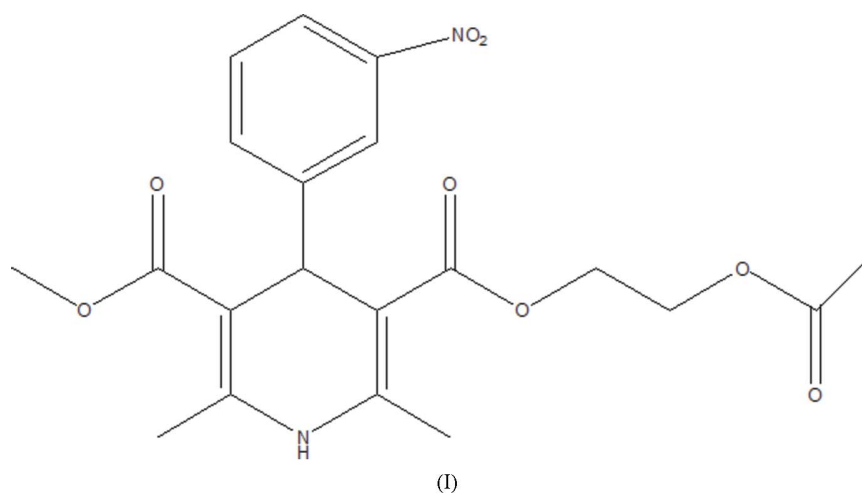


Fig. 1 shows the structure of (I). The dihydropyridine ring has a flattened boat conformation. This compares well with the structures of two 3-benzotriazol-1-yl 5-alkyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylates (Liu *et al.*, 2006; Jiang & Sun, 2006). Atoms C3 and N1 are displaced from the mean plane formed by the other atoms in the ring by 0.296 (1) and 0.130 (1) Å, respectively. The dihedral angle between the benzene ring and the C1/C2/C4/C5 plane is 94.54 (1)°. Intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules along the c axis (Table 1).

Experimental

3-(2-Hydroxyethyl) 5-methyl 2,6-dimethyl-4-(3-nitrophenyl)-1,4-dihydropyridine-3,5-dicarboxylate (376 mg, 1 mmol) and NaHSO_4 (22 mg, 0.2 mmol) were dissolved in ethyl acetate (15 ml). Acetic anhydride (5 ml) was added dropwise to the solution at room temperature. The reaction mixture was stirred at 300 K for a further 6 h. Water (20 ml) was added to the solution and the mixture was neutralized by NaOH (1 mol l^{-1}). The organic layer contained the

desired compound. The product was obtained by removing the solvent, ethyl acetate, by vacuum evaporation at 293 K, and purifying by chromatography on a silica-gel column (eluting with ethyl acetate and petroleum, 1:5) at room temperature. Suitable crystals were obtained by slow evaporation of an ethyl acetate–petroleum (1:6) solution.

Crystal data

$C_{20}H_{22}N_2O_8$ $V = 1002.8 (2) \text{ \AA}^3$
 $M_r = 418.40$ $Z = 2$
 Triclinic, $P\bar{1}$ $D_x = 1.386 \text{ Mg m}^{-3}$
 $a = 9.0389 (12) \text{ \AA}$ $\text{Mo } K\alpha \text{ radiation}$
 $b = 10.6990 (14) \text{ \AA}$ $\mu = 0.11 \text{ mm}^{-1}$
 $c = 11.0049 (16) \text{ \AA}$ $T = 113 (2) \text{ K}$
 $\alpha = 93.452 (6)^\circ$ Block, yellow
 $\beta = 90.871 (5)^\circ$ $0.32 \times 0.30 \times 0.24 \text{ mm}$
 $\gamma = 109.153 (6)^\circ$

Data collection

Rigaku Saturn diffractometer 12582 measured reflections
 ω scans 4730 independent reflections
 Absorption correction: multi-scan 3265 reflections with $I > 2\sigma(I)$
 (Jacobson, 1998) $R_{\text{int}} = 0.032$
 $T_{\text{min}} = 0.966$, $T_{\text{max}} = 0.975$ $\theta_{\text{max}} = 27.8^\circ$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0614P)^2]$
 $R[F^2 > 2\sigma(F^2)] = 0.038$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.109$ $(\Delta\rho)_{\text{max}} = 0.002$
 $S = 1.06$ $\Delta\rho_{\text{max}} = 0.42 \text{ e \AA}^{-3}$
 4730 reflections $\Delta\rho_{\text{min}} = -0.25 \text{ e \AA}^{-3}$
 281 parameters Extinction correction: *SHELXL97*
 H atoms treated by a mixture of Extinction coefficient: 0.032 (5)
 independent and constrained
 refinement

Table 1 Selected geometric parameters (\AA , $^\circ$).

N1–C5	1.3837 (17)	N1–C1	1.3881 (16)
C5–N1–C1	123.66 (11)	C4–C3–C2	110.44 (10)
C2–C1–N1	118.69 (12)	C4–C5–N1	119.15 (11)

Table 2 Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1–H1 \cdots O6 ⁱ	0.893 (16)	2.038 (17)	2.9241 (15)	171.7 (15)

Symmetry code: (i) $x, y, z + 1$.

H atoms on C atoms were placed in calculated positions and constrained to ride on their parent atoms, with $C-H = 0.93\text{--}0.98 \text{ \AA}$

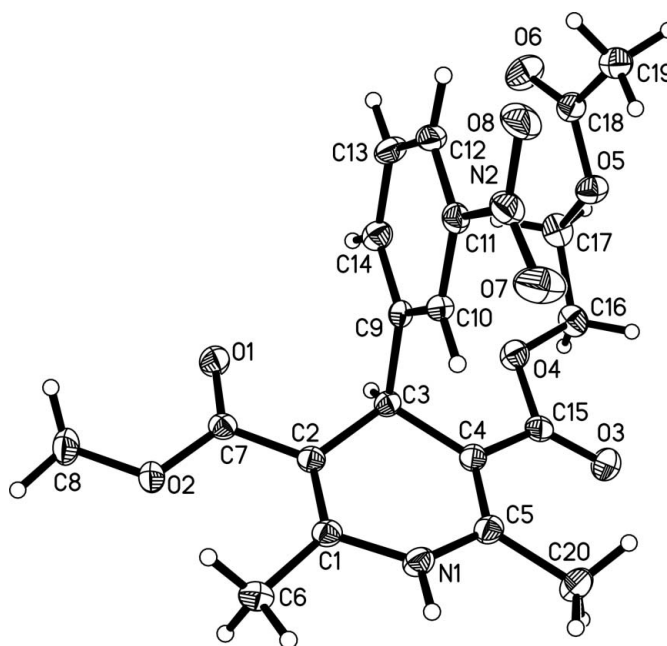


Figure 1 The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$. Atom H1 on N1 was identified in a difference Fourier map and refined isotropically.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSK, 2005); software used to prepare material for publication: *CrystalStructure*.

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